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Second Quarterly Report

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for

CHARACTERIZATION OF NICKEL-CADMIUM ELECTRODES

1 October, 1963 - 1 January, 1964

Contract No. NAS5 - 3477

Prepared by

General Electric Company

Advanced Technology Laboratories
Schenectady, New York

for

Goddard Space Flight Center
Greenbelt, Maryland

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Schenectady, New York *N.Y.* *65.* *65.*

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SUMMARY

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The objective of this contract is to develop a method of analysis and characterization of the electrodes used in nickel-cadmium sealed cells. It is based primarily on a comparison of detailed polarization measurements of single electrodes before and after periodic operation in selected modes of cyclic testing of cells at three temperature levels 0°, 25°, and 50° C. A correlation of this data should provide a basis for specifying improved cells for space applications as well as comparing cells from various manufacturers.

During the second quarter, the construction of the control and cyclic testing equipment required for the program was completed, and the equipment was checked out and debugged. Electrodes for the program were electrochemically cleaned, weighed, and stamped with identification numbers. Two batches of electrodes were put through the initial characterization tests and one cell was put on cyclic testing to investigate memory effects. The start of the remaining two sets of cyclic tests was delayed by modifications required in some of the control equipment and the need to investigate the cause of positive electrode blistering when characterized at charge and discharge rates of 1.0 amperes (nominal 1C rate). *Author*

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1.0 INTRODUCTION

This report covers the work done during the second quarter of an 18 month program to develop a method for the analysis and characterization of the electrodes used in nickel-cadmium spacecraft batteries.

The goal of the program is to develop a correlation between detailed characterization data obtained on single electrodes and the behavior of these electrodes in cells in various modes of cyclic operation. Such a correlation will provide a basis for specifying improved cells for space application and comparing cells from various manufacturers.

A breakdown of the program into tasks is given in Table I. The first three tasks are of a preparative nature. Tasks IV through VII constitute the experimental portion of the test program.

The test program is divided into two parts: one, the initial characterization testing of plates (Task IV); and the other, the cycling of cells made from characterized plates in selected modes of operation (Tasks V - VII). Periodically test cells will be removed from tests and the individual electrodes will be recharacterized and examined for changes in physical properties and comparisons made to the original characterization data.

The initial characterization will be made by analysis of data taken in single electrode experiments based on the use of continuous recording of charge-discharge curves under various testing regimes. The characterization information will include: 1) polarization of each electrode under various conditions, 2) complete charge and discharge curves showing electrode capacity, impurity levels, onset of gassing, graphitic and antipolar capacity, and reproducibility of cell operation, and 3) the onset of changes in capacity under various operating conditions.

All plates in the program will be SAFT type VO, prepared as for space cells. Tests will be made at three temperatures: 0°, 25°, and 50° C. as listed in Table II. Failure analysis on cycled cells will be made, using visual, mechanical, chemical and electrochemical procedures.

During the second quarter construction of the control and testing equipment for the characterization and cycling tests was completed and operation of the equipment was checked out. Sufficient electrodes for use in the test program were electrochemically cleaned, weighed and identified as described under Task III. In addition a total of 20 plates were characterized as per Task IV and the shallow cycling tests (Task V) were started at 25° C.

TABLE I
PROGRAM TASKS
NAS5 - 3477

TASK		MEASUREMENTS
I	Control and Recording Equipment Design and fabrication of test equipment.	None.
II	Test Cell Assemblies Design and fabrication of test cells.	None.
III	Electrode Preparation Electrochemical cleaning of reference and cell electrodes, inspection and welding of identification tabs.	Capacity check and weight.
IV	Characterization Tests - CA and CB C-A Constant current charging at C/10, C/5 and C C-B Constant current discharging at C/10, C/5 and C	C-A Determine charge curve. Determine rate of gassing from electrodes. Determine rate of O ₂ recombination. C-B Determine discharge curve.
V	Shallow Discharge Cycling Tests C-C Constant current cycling to 25-35% range to determine memory effects	C-C Make periodic capacity determination. Make analysis of physical properties. Recovery test.
VI	Random Discharge Tests R-A Random discharges averaging 10%, 25%, 50% and 75% depth of discharge over a 6-day period using Gaussian and rectangular distribution for discharge periods.	R-A Periodic charge and discharge curve. Recharacterization tests.
VII	Constant Voltage, Current Limi- ted Charging Cycling Tests R-B Charge at C/5 rate and discharge at C/2 rate to 0.9 volts.	R-B Periodic charge and discharge curve. Recharacterization tests.

TABLE II
TEST CELL SUMMARY

Test	Temperature			Total Cells	Plates/Cell				Ref.
	0°C	25°C	50°C		Ni Test	Cd Test	Ni Counter	Cd Counter	
CA-CB									
Characterization	8	8	8	24	1	1	2	2	1
Gassing	2	2	2	6	1	1	2	2	1
O ₂ Recombination	1	1	1	3	2	1	0	0	1
C-C	3	3	3	9	4	4	0	1	0
R-A				18	1	1	0	1	0
10%	1	1	1						
25% G	1	1	1						
25% S	1	1	1						
50% G	1	1	1						
50% S	1	1	1						
75%	1	1	1						
R-B	2	2	2	6	4	4	0	1	

2.0 DISCUSSION

The work performed on each task listed in Table I is reported in the following sections.

2.1 Control and Recording Equipment - Task I

The construction of the control equipment for conducting the CA-CB, C-C, R-A, and R-B tests was completed. The circuit schematics for these may be found in Appendix I of the first quarterly report.

Several problems were encountered in the CA-CB equipment which delayed the start of the characterization tests. Some of the Rustrak Recorders used to record the potential of the nickel and cadmium electrodes versus the reference electrode yielded erratic voltage readings. Apparently this was caused by ground loop potentials, existing between control panel circuits and the recorder amplifier chassis. Not all of the recorders behaved in this fashion when installed in the equipment cabinets. Calibration of these units removed from the cabinets showed them to be satisfactory. The problem was resolved by selecting amplifier units from stock that gave correct readings when installed in the cabinets.

The other problem involved a design error which tied the reference electrodes in the nickel and cadmium characterization test cells to a common electrical connection. Since the two test cells are connected in parallel to the charge and discharge power supply, connecting the reference electrodes together allowed them to discharge against the cadmium electrodes. The circuits were modified to break the common connection and switch the reference electrodes by means of additional relays actuated by the control timer. A revised circuit diagram showing these modifications may be found in Appendix I.

2.2 Test Cell Assemblies - Task II

No work was scheduled on this task for this quarter. The design and construction of the special test cells for determining gassing and recombination rates will be undertaken during the third quarter.

2.3 Electrode Preparation - Task III

Approximately 600 electrodes were electrochemically cleaned by the procedure described in Table II of the first quarterly report. Electrodes rejected on the basis of visual defects were less than one percent.

The final weights of the electrodes after cleaning are tabulated in Appendix II. These will be reweighed prior to characterization to provide a reference point for comparison after the electrodes are removed from the

various cyclic tests. The average weight of the positive elements after cleaning was 11.150 gms with a standard deviation of 0.290 gms, and for the negative electrodes the average was 10.161 gms with a standard deviation of 0.259 gms. Weight changes for the positive electrodes before and after cleaning averaged ± 0.028 gms; for the negatives the weight decreased on the average of 0.273 gms.

2.4 CA-CB Tests - Task IV

Two batches of electrodes (10 positives and 10 negatives per batch) were characterized at nominal C/5 and C rates, at room temperature (approximately 25° C.). The cycle conditions for the C/5 rate tests were, charge for 360 minutes and discharge for 300 minutes at 0.28 amp. For the C rate tests, the conditions were charge for 101 minutes and discharge for 84 minutes at 1.0 amp. In both cases the charging time was approximately 120% of the discharge time.

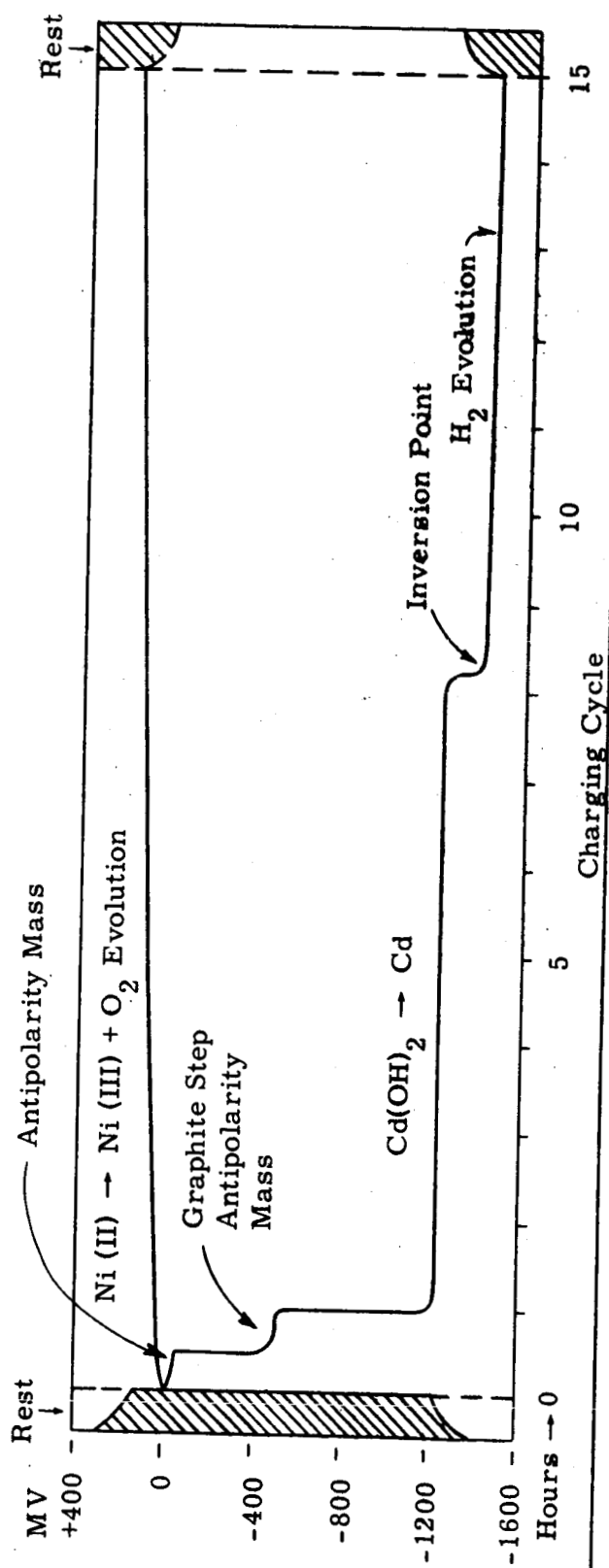
All plates were given seven consecutive complete charge and discharges. Potential time curves for each electrode versus a fully charged and aged nickel reference electrode were continuously recorded for the seven cycles. The potential time curves at the C/5 rate for both positive and negative electrodes were observed to be normal with respect to the general shapes of the curves and characteristic points of inflection at various stages in the charge-discharge cycle. The types of curves observed are shown in Figure 1.

The most noticeable trends observed over these initial cycles were: for the nickel electrode, a progressive increase in the length of the graphite step in the discharge portion of the cycles; for the cadmium electrode, a progressive decrease in time at which the inversion point was obtained. This behavior is not unusual and has been observed in previous experiments. The full significance of these changes and interpretation must await the results of studies which will be made on the electrodes after removal from the various cycling tests later in the program.

The nickel electrodes characterized at the C rate were observed to develop blisters on both surfaces in which the sintered nickel matrix was separated from the nickel-plated sheet of the electrode. Examples of this phenomenon are shown in the photograph of Figure 2. Nine out of ten of the nickel electrodes in the batch showed blisters ranging from approximately 1/8 to 1/2 in. in diameter.

Supporting studies are being undertaken to determine if the phenomenon is the result of a defective lot of electrodes or whether the electrode is being subjected to gassing conditions which cannot be accommodated by the electrode structure.

A check with the Battery Product Section at Gainesville, Florida, was



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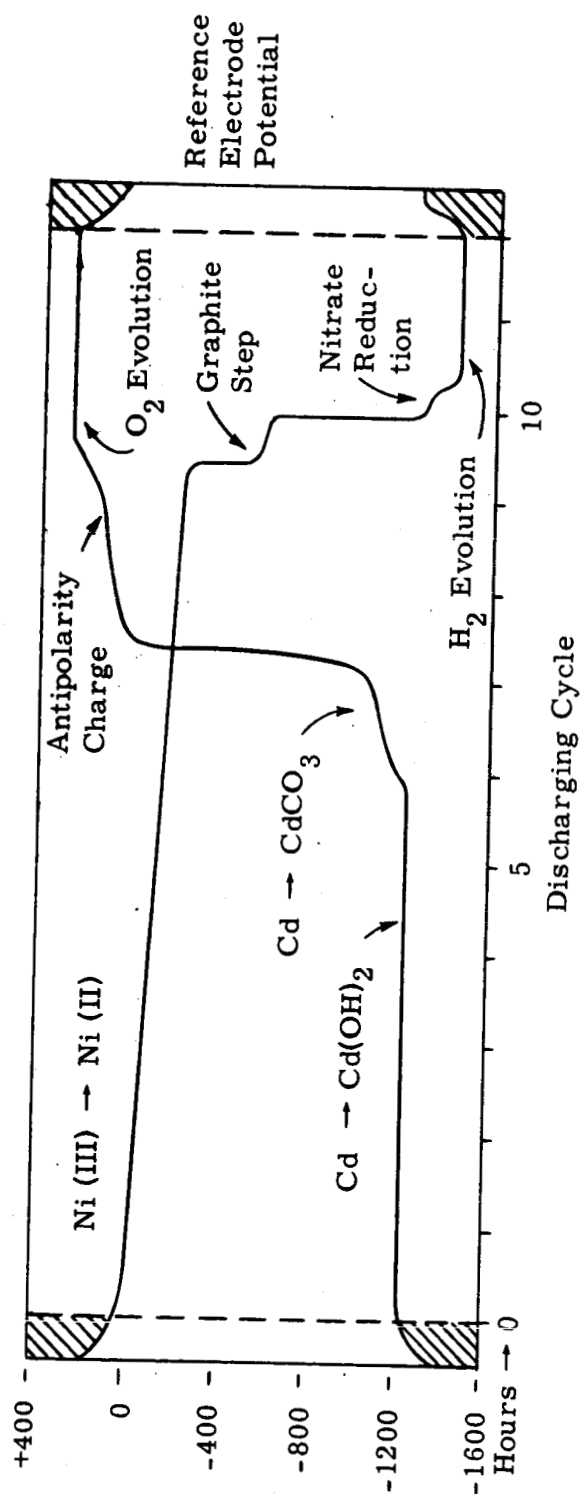


Figure 1. Typical Characterization Test Curves.

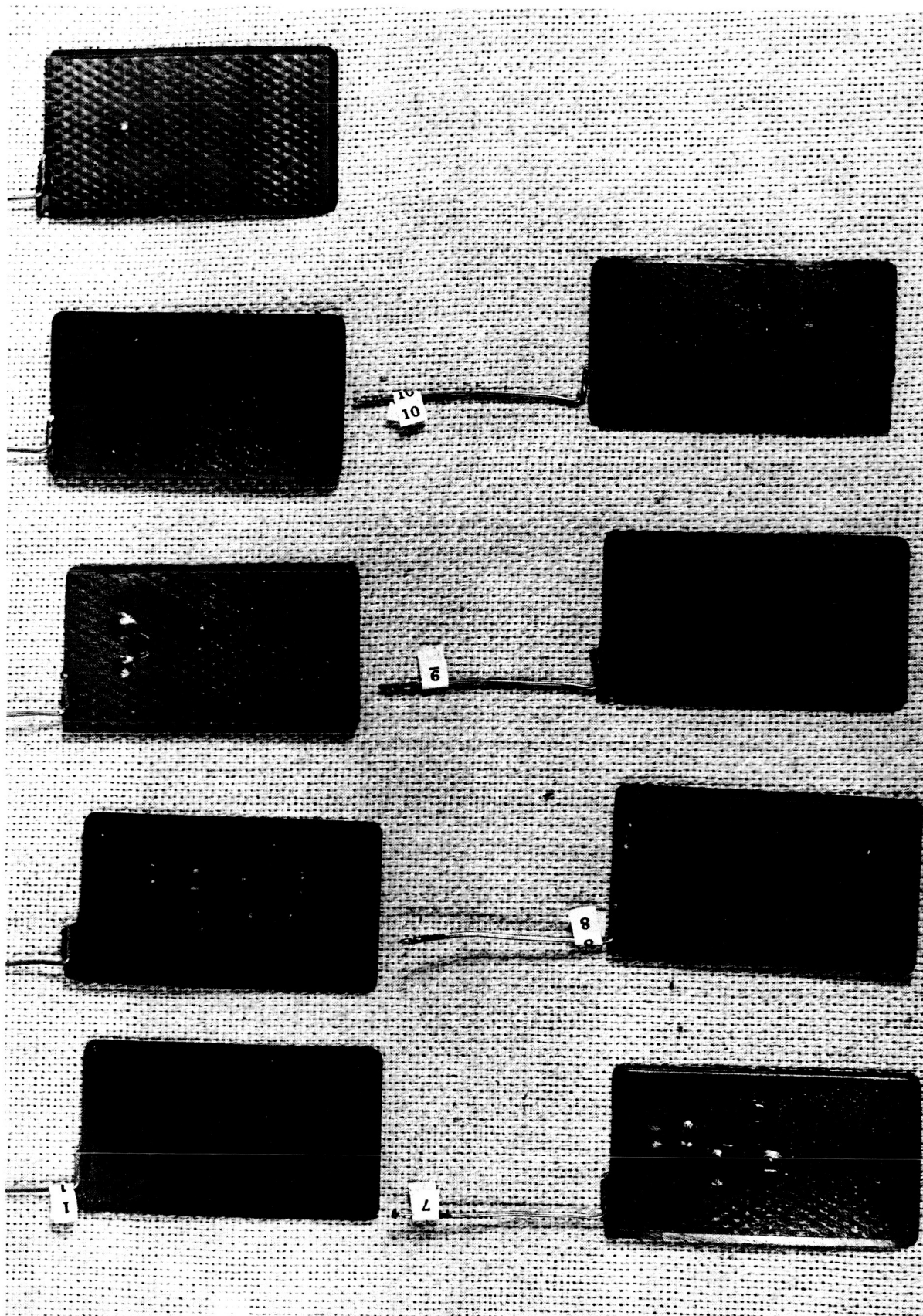


Figure 2. Positive Electrode Blistering - Nominal Plate Capacity 1.0 Amp-Hr.
Conditions - Charge 101 min, Discharge - 84 min @ 1.0 Amp - 25°C.

made with respect to history of this lot. There was no history of manufacturing problems at the time the plaques were made. However, the electrodes had not been used in any cells and therefore there was no comparative information as to their performance in cells. A less severe case of this phenomenon has been observed at Gainesville when nickel electrodes were run in the inverted conditions at rates of approximately C/8 to C/5 for periods of twenty to twenty-four hours. It may be that at the C rate the phenomenon may be more severe in a short period of time. The time that the electrodes were in the inversion mode in the cycle used was approximately 15 minutes. In addition, an examination of the potential time curve shows an inflection point for the nickel electrode prior to the end charge which is an indication of heavy gassing. Such an inflection was not observed for the electrodes run at C/10 or C/5 rates. Thus it may well be that the combined gassing on charge and on over-discharge may both be contributing to the blistering.

Pending the results of the supporting studies which will be completed in the third quarter, it is planned to restrict all future initial characterization tests to the C/10 rate.

2.5 C-C Tests - Task V

These tests are designed to determine the onset and severity of memory effects. Test cells consisting of five cadmium and four nickel electrodes per cell will be cycled at constant current simulating the conditions of a 90 minute orbit, 35 minute discharge and 55 minute charge. The depth of discharge will be limited to 25%.

One cell was put on test during the quarter. It is planned to cycle this one for 500 to 600 cycles and then check one pair of electrodes for capacity using fully charged counter electrodes to 0.9 volt at the cycling discharge rate, followed by discharge through a resistor to zero voltage. This will check the capacity of the plate and determine which plate is showing the memory effect and the severity of the effect.

If memory has set in, a test plate will be removed without discharge and examined by physical methods (X-ray diffraction, etc.) to determine what changes if any have occurred. The remaining plates will be electrochemically tested to determine recovery from memory and recharacterized plates will be replaced in the cycling to accumulate data.

2.6 R-A and R-B Tests - Tasks VI and VII

The problems encountered in the previous tasks delayed the start of these tests. The testing equipment required, as indicated earlier, is completed and available for use.

During the next quarter as a sufficient number of plates are accumulated from the initial characterization tests, cells will be put on cyclic testing as described in the first quarterly report.

3.0 PROGRAM FOR NEXT QUARTER

The characterization test CA-CB for electrodes to be used in the cyclic tests will continue using the C/10 rates (~ 0.1 amp.) for all electrodes. Supporting studies on the blistering problem will be conducted to determine the cause of this phenomenon.

As electrodes are accumulated from the CA-CB tests, cells will be put on cyclic testing. The rate at which these tests can be gotten underway will be slower than originally scheduled since more time will be required for the characterization at the C/10 rate. The characterization time at this rate is approximately 4 1/2 days per batch of 10 positives and 10 negatives, allowing for setup time for each batch.

These changes will reduce the total number of cyclic test data that can be accumulated on each task. However, it will not affect the achievement of the overall program objectives; namely, to identify the importance of the factors being investigated in each task.

4.0 NEW TECHNOLOGY REPORT

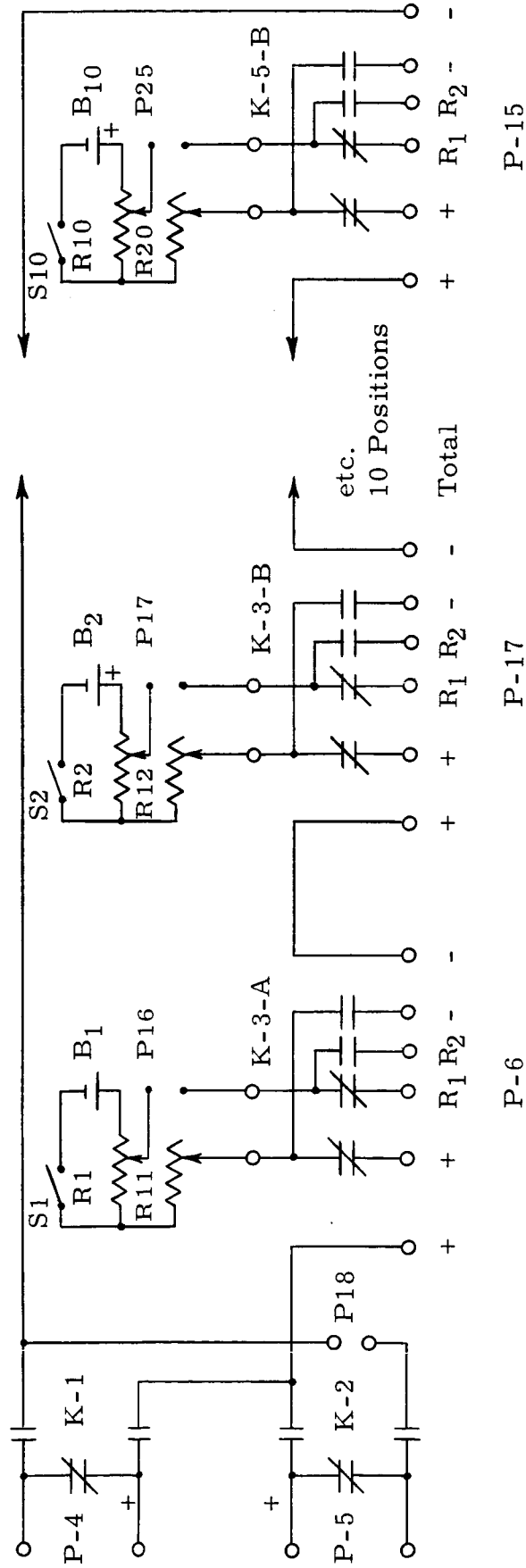
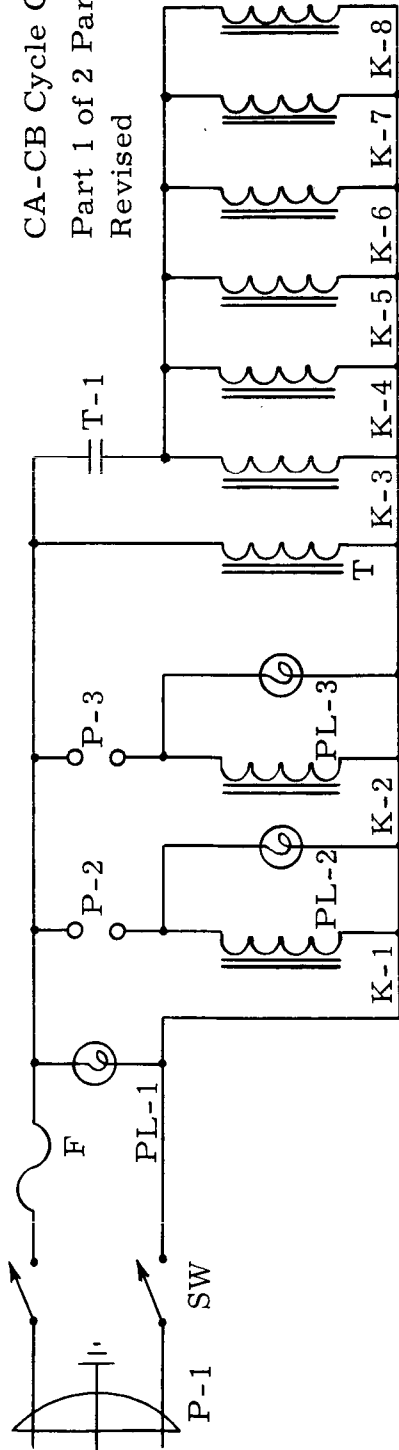
There were no new developments during this quarter which come under the "New Technology" clause of this contract.

APPENDIX I

Control Equipment Schematics

Ni-Cd Characterization

CA-CB Cycle Control Part 1 of 2 Parts Revised



Note: RL changed with variation in load.

C/10	500	12.5 Watts	Ohmite 0117
C/2	100	25	Ohmite 0151
C	50	50	Ohmite 0318

PARTS LIST

Part 2 of 2 Parts

P-1 Line Cord, CA-CB Controller
P-2 Control, Charge, Pin Jacks, Yellow
P-3 Control, Discharge, Pin Jacks, Red
P-4 Input Charge, CCS, Terminal
P-5 Input Discharge, CCS Terminal
P-6 thru P-15 Pin Jacks, Cell Input Red +, Green +, White R, Blue -, Black -
P-16 thru P-17 Pin Jacks Recorder, Red +, Black -
P-18 Pin Jacks, G. R. for RL
SW Switch, Toggle SPDT
F Fuse, 3A
PL-1 Pilot, Master, Green
PL-2 Pilot, Charge, Yellow
PL-3 Pilot, Discharge, Red
T Timer, CM, 2 Min
RL Load Resistance (See Drawing)
K-1, K-2 Mercury Relay, 3PST 2NO, 1NC, Ebert EM7, 110 VAC Coil
K-3, K-4 Leach 329-7 4PDT, 110 VAC Coil
K-5, K-7, K-8 Leach 337 2PDT, 110 VAC Coil
S-1 thru S-10 Switch SPST Toggle "Bias"
B-1 thru B-10 Battery Mercury O Cell
R-1 thru R-10, 50,000 Ω 2 Watt
R-11 thru R-20, 3 Meg

APPENDIX II

Weights of Electrochemically Cleaned Electrodes

WEIGHT TABULATION OF ELECTROCHEMICALLY CLEANED ELECTRODES

Prefix Number	Positive Electrodes				Negative Electrodes			
	PI	P3	P5	P7	NI	N3	N5	N7
Weight - grams								
Electrode Serial Number								
1	11.412	10.870	11.255*	11.041	9.450	9.884	10.544	9.923
2	11.389	11.094	11.509	11.185	10.546	10.391	9.852	10.062
3	10.911	10.932*	11.445	11.013	10.547	9.975	10.202	9.683
4	11.262	11.329	11.231	10.749	9.943	9.969	10.045	10.552
5	11.251	11.506*	11.198	11.730	9.806	10.472	9.971	10.00
6	11.517	10.928	11.497	10.851	10.318	9.912	10.436	9.80
7	11.112	11.564	10.931	10.721	9.853	9.851	9.824	10.23
8	10.838	10.749	10.821	10.853	9.655	10.474	10.344	10.50
9	10.759	10.905	10.973*	10.733	10.197	10.059	9.999	9.50
10	10.758	10.905	10.803	11.551	9.868	10.499	9.953	10.50
11	11.032	11.454	10.830	11.176	10.199	9.852	10.200	10.00
12	10.890	11.109	10.786*	10.86	9.672	9.940	10.280	10.60
13	11.326	10.985	11.532	11.13	10.506	10.628	10.107	"none"
14	10.725	11.094	11.200	11.44	9.975	9.978	9.923	
15	11.010	11.406	11.146	11.23	9.939	10.391	10.062	
16	11.343	11.406	10.751	11.13	10.200	9.653	10.561	
17	11.542	11.046	11.139	11.00	10.349	10.087	9.967	
18	11.090	11.093	11.140	11.13	9.966	10.586	10.053	

* Defective

WEIGHT TABULATION CONT'D

Prefix Number	Positive Electrodes				Negative Electrodes			
	PI	Weight - grams P3	P5	P7	NI	Weight - grams N3	N5	N7
19	11.048	11.025	11.937	"none"	10.410	10.294	10.660	"none"
20	10.850	10.840	10.753*		10.263	9.954	9.822	
21	11.206	11.424	10.824*		10.322	10.136	10.512	
22	11.152	10.829*	11.088*		10.278	10.143	9.795	
23	11.081	11.283	11.324		10.290*	10.363	9.937	
24	10.699	11.392	10.996		9.959	10.236	9.671	
25	10.906	10.760	11.039		10.254	10.357	10.131	
26	10.699	"none"	11.002		10.634	10.549	9.830	
27	10.908		10.842		9.805	9.889	10.523	
28	12.099		11.393		10.589	10.088	9.972	
29	11.036		11.142		10.383	10.054	10.503	
30	11.290		11.383		10.134	10.585	10.158	
31	10.796		"none"		10.005	10.241	10.217	
32	10.799				10.498	10.598	10.205	
33	11.797				10.514	10.426	10.364	
34	11.540				10.618	9.986	9.895	
35	10.703				9.782	9.997	10.015	
36	10.929				10.036	10.391	"none"	

*Defective

WEIGHT TABULATION CONT'D

Prefix Number	Positive Electrodes Weight - grams				Negative Electrodes Weight - grams			
	PI	P3	P5	P7	NI	N3	N5	N7
Electrode Serial Number								
37	11.067	"none"	"none"	"none"	10.557	10.441	"none"	"none"
38	11.582				10.71	10.170		
39	11.322				9.987	10.079		
40	10.866				10.318	10.123		
41	11.598				10.406	10.143		
42	10.837				10.227	10.109		
43	10.886				9.838	10.110		
44	11.896				10.436	9.946		
45	11.415				9.876	10.108		
46	11.175				10.309	10.031		
47	11.291				10.109	10.305		
48	11.455				10.192	10.220		
49	11.175				10.137	10.461		
50	11.493				10.121	10.497		
51	10.721				10.189	"none"		
52	11.411				10.465			
53	11.244				9.927			
54	11.527				10.503			

WEIGHT TABULATION CONT'D

Prefix Number	Positive Electrodes Weight - grams				Negative Electrodes Weight - grams			
	PI	P3	P5	P7	NI	N3	N5	N7
Electrode Serial Number								
55	10.889	"none"	"none"	"none"	10.002	"none"	"none"	"none"
56	11.221				9.860			
57	11.240				10.222			
58	11.128				10.461			
59	10.977				9.898			
60	11.235				10.322			
61	11.442				10.372			
62	11.492				10.215			
63	11.270				9.916			
64	11.521				10.251			
65	11.284				9.895			
66	11.315				9.876			
67	11.086				10.289			
68	11.274				10.106			
69	11.098				10.237			
70	11.055				10.006			
71	11.532				9.877			
72	10.987				10.380			

WEIGHT TABULATION CONT'D

Prefix Number	Positive Electrodes Weight - grams				Negative Electrodes Weight - grams			
	PI	P3	P5	P7	NI	N3	N5	N7
Electrode Serial Number								
73	11.019	"none"	"none"	"none"	9.812	"none"	"none"	"none"
74	10.902				10.204			
75	10.892				10.396			
76	10.943				9.559			
77	10.911				10.252			
78	10.703				9.960			
79	10.960				10.131			
80	11.227				9.934			
81	10.697				10.370			
82	10.779				10.320			
83	11.387				10.011			
84	11.466				10.380			
85	11.349				10.326			
86	11.512				10.175			
87	11.943				9.914			
88	11.324				10.013			
89	10.891				10.032			
90	11.219				10.504			

WEIGHT TABULATION CONT'D

Prefix Number	Positive Electrodes Weight - grams				Negative Electrodes Weight - grams			
	PI	P3	P5	P7	NI	N3	N5	N7
Electrode Serial Number								
91	11.606	"none"	"none"	"none"	10.156	"none"	"none"	"none"
92	11.576				10.311			
93	10.819				10.064			
94	11.411				10.159			
95	11.182				10.035			
96	11.486				10.400			
97	11.421				10.416			
98	11.032				9.755			
99	11.132				10.227			